Table 6 List of solvents for separation of morphine

Solvent	*CAS-NO*	*Predicted*			*Experimental*			*Compound class (RTECS)*
		T_b (K)	T_m (K)	δ (MPa$^{1/2}$)	T_b (K)	T_m (K)	δ (MPa$^{1/2}$)	
Benzene	71-43-2				353.24	278.68	18.73	C,D,M,T,S
Toluene	108-88-3				383.78	178.18	18.32	C,M,T,S
CCl_4	56-23-5				349.79	250.33	17.55	C,D,M,T,S
Cyclohexane	110-82-7				353.87	279.69	16.76	M,S
1,5-Pentanediol	111-29-5	491	253	27.0	512.15	257.15	26.45	S
Acetol	116-09-6	418	226	27.2	418.65	256.15	25.75	M

D, drug; S, primary irritant; T, reproductive-effector; M, mutagen; C, tumorigen.

however, need to integrate aspects of molecular modelling and computational chemistry before acceptable solutions to problems involving complex solutes and tight environmental regulations can be obtained. Finally, it should be noted that having a good solvent means easier design/operation of the solvent-based separation technique. Therefore, it is important to formulate correctly the solvent selection problem and to find reliable results in the form of optimal solvents.

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Steam Distillation

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Sample preparation is nowadays the limiting step in the trace analysis of organic pollutants in environmental and biological samples. Looking forward to the laboratory of the future, versatile and universal sample enrichment techniques are required, which can produce fast and valid data, with low costs in terms of solvent consumption and operator involvement. A selectivity higher than that of the classical exhaustive extraction methods or the simultaneous elimination of the interference material could be an additional requirement, as it would reduce the amount of solvents and adsorbents used by reducing or eliminating the subsequent clean-up step. Possible additional benefits deriving from a low manual manipulation of the samples would be a reduction in the risk of contamination and loss of the analytes, as well as an easier automation of the process.

Steam distillation extraction–solvent extraction (SDE) has been presented as such a universal sample enrichment technique. SDE allows the simultaneous extraction, clean-up and concentration of the target compounds in a closed system, with short analysis times (1–8 h) and by using small amounts of organic solvents (a few mL). This paper reviews this assumption for the case of the analysis of less volatile organic pollutants in environmental samples. The SDE advantages and shortcomings for such an analysis have been discussed.

Introduction

The monitoring of toxic organic chemicals in environmental and biological samples is a major concern in many different fields. However, the large variety of compounds of interest, the differences existing in

their environmental levels and physico-chemical properties, and the complexity of the matrices typically investigated make the development of universal analytical methods for such an analysis a very difficult goal. This is especially true for the most toxic organic pollutants as their high toxicity makes their reliable detection and accurate quantification at the trace level more relevant.

Most of the procedures described in the literature for the analysis of less volatile organic pollutants are time-consuming, laborious and specific for the determination of an analyte (or family of compounds) in a selected matrix. Examples of selective extraction of the target compounds, allowing their determination without any additional clean-up, can be found in the literature. However, most of these procedures involve sophisticated and expensive analytical techniques, such as supercritical fluid extraction or gel permeation chromatography. On the other hand, the efficiency of these methods have been recognized to be highly matrix-dependent. Because of these unresolved shortcomings, classical exhaustive extraction techniques, i.e. liquid–liquid extraction, LLE, solid–liquid extraction or Soxhlet extraction, are still widely used in official methods and routine applications. Due to the low selectivity of these methods, subsequent elimination of the co-extracted material is recommended. Such a clean-up step is mandatory for reliable trace level determination of lipophilic and bio-accumulative pollutants in biological and complex environmental samples.

Steam distillation-solvent extraction (SDE) has been used mainly for the extraction and concentration of fragrance and flavour compounds. However, a variety of SDE methods reporting sample preparation for the analysis of pollutants in environmental samples can be found in the literature. Most of these methods allow the simultaneous extraction, clean up and concentration of the target compounds. The investigated compounds range from volatile polar and non-polar pollutants to non-ionic surfactants. This article reviews the suitability and the limitations of SDE for the determination of less volatile trace organic pollutants, such as pesticides, polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs), or surfactants, in environmental and biological matrices. The most relevant variables affecting the efficiency of the SDE of these compounds are discussed and the results of some selected applications reviewed.

General Considerations

According to the theoretical model developed by Rijks *et al.*, in 1983, the efficiency of the SDE process increased with the extraction time and with the liquid and vapour flows. The process also depends on analyte-specific parameters related to the activity coefficient (calculated from the water solubility of the analyte at 100°C) and the gas-liquid distribution coefficient of the compound in water at the process temperature (i.e., 100°C for water steam). Not unexpectedly, the recoveries increased with the affinity of the target compounds for the extracting solvent. This theoretical model is applicable only under ideal conditions, which are achieved when all volumes and flow rates remain constant and there is ideal mixing and equilibrium at every stage. In spite of these limitations, the model reflects the effect of several experimental factors on the SDE process. In fact, the different modifications carried out on the SDE devices originally described by Likens and Nickerson in 1964 and by Flath and Forrey in 1977 reveal the influence of several parameters on the recoveries of the target compounds. The modifications were mainly focused on increasing the size of the vapour chamber and/or the condensing surface to allow a more complete mixing of the solvent and steam vapours, as well as on the miniaturization of the system. As a consequence of the changes in design (**Figure 1**), the efficiency of the extraction was increased, the analysis time reduced and the field of SDE expanded through the analysis of residue levels of less volatile pollutants in environmental samples.

Due to the characteristics of the technique, the feasibility of SDE for the analysis of less volatile compounds depends on their (i) potential for forming azeotropes with water and (ii) relative solubility in water and in the extraction solvent. However, the SDE of the target compounds from complex samples can be expected to occur only after destruction or degradation of the main matrix components, which usually entrap the analytes (see below). Therefore, as stated by Nash in 1984, the applicability of the SDE technique to the analysis of this kind of environmental matrices would be limited by the resistance of the investigated compounds to the selected degradation procedure. Alternatively, in some cases, co-distillation solvents have been used to improve the SDE efficiency by reducing the surface tension of the water and by increasing the extraction power (polarity) of the organic solvent. Finally, rather different results have been published about the suitability of adding anti-foam agents in applications involving fatty samples (see **Table 3**).

Application of SDE to the Analysis of Aqueous Samples

Water was one of the first environmental samples selected to evaluate the feasibility of the SDE

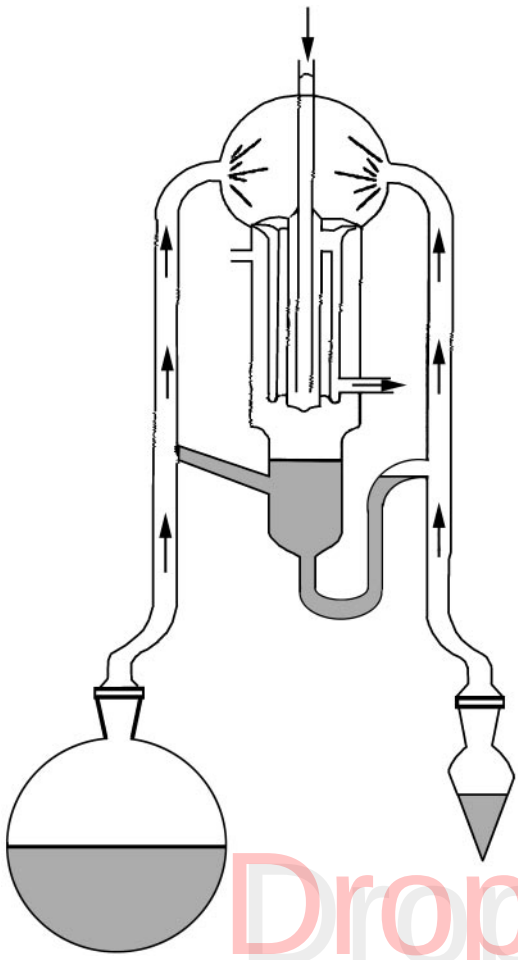

Figure 1 A typical SDE modern design.

technique for the determination of less volatile organic contaminants levels. **Table 1** summarizes relevant data concerning some reported methods for the analysis of this matrix.

Quantitative recoveries of spiked organochlorinated pesticides, OCPs (globally, in the range 90–106 ppb), and PCBs (globally, in the range 70–104 ppb) in aqueous samples have been reported using the SDE technique. The reported methods allowed the simultaneous extraction and concentration of the analytes in 1–1.3 h in a relatively small amount of a non-polar solvent (1–15 mL). Usually, no additional treatment of the sample or the organic extract was required. The SDE technique was favourably compared with other widely used extraction procedures, such as LLE or solid-phase extraction (SPE) by Ramos *et al.* in 1995, e.g. similar recoveries have been published for the analysis of PCBs in water at the ppb ($ng\,mL^{-1}$) level by using SDE, LLE or SPE. However, the higher repeatability of the SDE procedure (relative standard deviations, RSD, lower than 10%) and the small amount of organic solvent involved, as well as the short sample preparation times, makes SDE a valuable alternative technique for such an extraction, especially when a large number of analyses have to be carried out.

Nevertheless, some limitations of SDE have also been reported for less volatile pollutants in water samples. Nash *et al.* in 1984 studied different parameters affecting the efficiency of the steam distillation process. They concluded that this technique is probably limited to compounds with a vapour pressure of about 1 kPa at 100°C. Their results also showed that the performance of SDE depends on the concentration investigated and that recoveries tend to increase with the spiking level.

Similar tendencies have been observed by Ramos *et al.* in 1995 when using the SDE technique for the extraction of water spiked with the 2,3,7,8-substituted-CDD/Fs at different levels of concentration (0.25–2 $ng\,mL^{-1}$, 0.025–0.2 $ng\,mL^{-1}$ and 0.0025–0.02 $ng\,mL^{-1}$). The recoveries obtained for the lower and higher boiling point congeners (tetra- and octa-CDD/Fs, respectively) are consistently lower than those found for the rest of the investigated congeners: respectively 40–76% and 73–137% at the highest level of concentration investigated, 39–60% and 62–92% at the intermediate, and 37–55% and 25–72% at the lowest spiking level. These results also show that the SDE recoveries for a given compound decrease with the concentration level when using n-pentane as the extraction solvent. The simple substitution of n-pentane for a solvent more selective for the PCDD/Fs (dichloromethane) increases recoveries from 25–73% to 71–139% for tetra- to hepta-CDD/Fs at the 0.0025–0.02 $ng\,mL^{-1}$ level. However, no additional improvement is obtained for the octa-CDD/F recoveries (38–56%). In spite of the low recoveries obtained for OCDD/F, the proposed SDE procedure compares favourably with results previously published by using LLE or SPE in terms of repeatability, analysis time and solvent consumption.

Good recoveries (in the range 84–100%) have been reported by Meissner *et al.* for the analysis of surfactants such as fatty alcohol sulfates and alkyl polyglycosides in water (**Table 1**). SDE of the fatty alcohols yielded by hydrolysis and subsequent LLE of the original compounds is an attractive technique for the effective clean-up and concentration of these complex mixtures of compounds at the trace level. On the other hand, the application of SDE to the extraction of fatty alcohol ethoxylates with more than three ethoxy units in the molecule cannot be accomplished due to their high solubility in water.

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Table 1 SDE methods for the analysis of less volatile organic pollutants in aqueous samples

Compound	*Spiking level (ng mL^{-1})*	*Solvent (mL)*	*Extraction time (h)*	*Cc. factor[a] (water : solvent, v/v)*	*Post-treatment[b]*	*Recovery (%)*	*RSD (%)*	*Ref.*
OCP	0.004–0.016	Isooctane/toluene (15)	1	167 : 1	NR[c]	90–104	?	Hemmerling *et al.* (1991)
Arochlor 1016, 1242, 1248, 1254	0.016	Isooctane/toluene (15)	1	167 : 1	NR	98–100	?	
OCP	0.4–4.0	n-pentane (1)	1.3	50 : 1	NR	97–106	?	Godefroot *et al.* (1982)
Arochlor 1260	10	n-pentane (1)	1.3	50 : 1	NR	81–104[d]	?	
Toxic PCBs	0.01–1.0	n-pentane (2)	1	50 : 1	Concentration	70–115	<10	Ramos *et al.* (1995)
PCDD/Fs	0.025–2.0	n-pentane (2)	1	50 : 1	Concentration	49–139	<10	
PCDD/Fs	0.0025–0.02	Dichloromethane (2)	1	50 : 1	Change of solvent	49–139	<10	
Fatty alcohol sulfates	500 nM	Ethyl acetate (2)	3[e]	100 : 1	Derivatization	87–100	5.6–7.0	Meissner *et al.* (1999)
Alkyl poly-glycosides	2 μM	Ethyl acetate (2)	4[e]	25 : 2	Derivatization	84	?	

[a]Concentration factor.
[b]Post-SDE treatment required.
[c]NR, not required.
[d]Recoveries for some selected peaks.
[e]The SDE was conducted after hydrolysis with 4 M H_2SO_4 plus liquid–liquid extraction with diethyl ether of the hydrolysate and concentration.

Application of SDE to the Analysis of Non-Fatty Environmental Samples

Table 2 summarizes relevant data related to some reported SDE methods for the analysis of less volatile organic pollutants in non-fatty environmental samples. Most of the reported SDE applications referred to the analysis of OCPs and toxic aromatic compounds, e.g. PCBs, polychlorinated naphthalenes (PCNs), or polynuclear aromatic hydrocarbons (PNAs), in soils and sediments. Contrary to what might be expected from the high complexity of these samples, most of the methods did not include any further pre-treatment of the matrix but blending with the selected volume of water. Only a few procedures involving drastic treatments (e.g. blending of the sample with H_2SO_4 and $K_2Cr_2O_7$) during the SDE to guarantee the destruction of the soil or sediment components in which the target compounds could be entrapped, can be found in the literature.

The efficiency (or need) of such a drastic treatment is difficult to evaluate from the data published. In general, high (quantitative) recoveries have been reported for freshly spiked analytes (globally in the range 78–102% for PCBs and OCPs at the 20–90 μg g^{-1} level) with all the procedures (**Table 2**). However, the efficiency of the proposed SDE methods for the extraction of endogenous pollutants from weathered samples has been scarcely evaluated. In these studies, Seidel *et al.* (1993) and Cooke *et al.* (1980) found concentrations very close or below the limit of detection have usually been reported for the endogenous contaminants, but the lack of comparison of the SDE results with those obtained by standard or more exhaustive methods, i.e. Soxhlet extraction, do not allow any discussion about the methods used.

Dunnivant *et al.* in 1988 reported recoveries ranging from 47 to 99% for SDE of certified sediments with PCBs at the 2.34–24.6 μg g^{-1}. However, as quoted above, this SDE method involved a digestion of the sample under drastic conditions.

In a closely related study, Nash *et al.* compared the efficiency of steam distillation with subsequent organic solvent extraction to that of Soxhlet extraction for the analysis of pesticides in soil, plant tissues and air (polyurethane foam filters). Both procedures provided similar recoveries for the spiked samples (in the ranges 80–90%, 80–90% and 90–100%, respectively). However, the SDE levels determined for weathered soils blended with water were 40–50% lower than the concentrations found by the Soxhlet procedure. The study also showed that the efficiency

Table 2 SDE methods for the analysis of less volatile organic pollutants in non-fatty environmental samples (symbols as in Table 1)

Matrix	*Compound*	*Spiking level* ($\mu g\,g^{-1}$)	*Pre-treatment*	*Solvent*	*Extraction time (h)*	*Post-treatment*	*Recovery (%)*	*RSD (%)*	*Ref.*
Sediment	Arochlor 1016	50	Blended with 2.5 L water	Isooct/tol[a](15)	1	Elimination S	78	?	Veith *et al.* (1977)
Weathered sediment	PCBs, PCNs, PNAs	–	Blended with 0.8–1.1 L water	n-Hexane (10)	2.2	Concentration	–	1.1–23	Cooke *et al.* (1979)
Sediment	Chlorinated benzenes	100–1000	Blended with 0.25 L water	n-Hexane (10)	3	Elimination S	76–91	0.5–21	Onuska *et al.* (1985)
Sediment	Chlorinated benzenes	10–100	Blended with 0.25 L water	n-Hexane (10)	3	Elimination S	71–88	1.4–33	
Sediment	Chlorinated benzenes	1–10	Blended with 0.25 L water	n-Hexane (10)	3	Elimination S	66–89	1.4–17	
Certif. Sediment	PCBs	2.34–24.6	200 mL $H_2SO_4 + K_2Cr_2O_7$	n-Hexane (15)[b]	8	Alumina	47–99	0.3–5.4	Dunnivant *et al.* (1988)
Sediment	PCBs	33.6–90.0	200 mL $H_2SO_4 + K_2Cr_2O_7$	n-Hexane (15)[b]	8	Alumina	102	4.7–9.3	
Soil	HCB	20.0	100 g soil + 20 mL water + 10 mL ethanol + ultrasonic, 1 min	Petrolbenzine (?)	1	NR	100	2.7	Seidel *et al.* (1993)
Weathered soil	Endogenous OCP	–	100 g soil + 20 mL water + 10 mL ethanol + ultrasonic, 1 min	Petrolbenzine (?)	1	NR	–	–	
Particulate	PCDD (homologues)	0.045–8.56[c]	150 g (sample + water) + HCl + sonication	n-Hexane (10)	3	Basic alumina	85–116[d]	?	Townsend *et al.* (1989)
Fruits, vegetables	OCCs, OCPs	0.01–1.0	5–10 g sample blended with 0.25 L water	n-Hexane (5)	1.5	NR	66–108	?	Hemmerling *et al.* (1991)
Fruits, vegetables	PCBs	0.1	5–10 g sample blended with 0.25 L water	n-Hexane (5)	1.5	NR	68–89	?	
Sediments	Chiral PCBs	–	5 g sample blended with 4 g Cu + 50 mL water	n-Pentane (2)	1	NR	–	–	Glausch *et al.* (1996)
Sewage sludge	Nonylphenol polyethoxylates	100[e]	1 g sample blended with 0.1 L water	Cyclohexane (1–2)	3	NR (HPLC) Alumina (GC)	< 30[f]	?	Lee *et al.* (1997)
Toothpaste	Fatty alcohol sulfates	–	30 g sample + 50 mL 4M H_2SO_4 + LLE (diethyl ether, 10 mL) + concentrat.	Ethyl acetate (2)	3	Derivatization	–	–	Meissner *et al.* (1999)

[a]Isooctane/toluene.
[b]Replace hexane layer after 1, 2, 4 and 8 h intervals.
[c]Range for different homologues.
[d]Mass balance calculations for the whole SDE system by comparison with the initial amount.
[e]Total concentration for all NP*n*EO ($n = 1$–17).
[f]By comparison with SFE, except for NP1EO.

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of the SDE depends on the soil type and, in agreement with that mentioned for aqueous samples, on the volatility of the selected compounds. The less volatile the compound, the lower the recovery: SDE recoveries for DDT were only 21–60% of those found by Soxhlet extraction.

Onuska and Terry observed a similar trend when comparing the SDE and the Soxhlet efficiencies for the extraction of spiked chlorinated benzenes from a sediment. The concentrations found using SDE were 14–36% lower than those using the Soxhlet method, except for pentachlorobenzene and 1,3-dichlorobenzene, which were not determined by the latter procedure. The authors also reported a decrease in the SDE recoveries of the target compounds as the investigated concentration level decreased (Table 2). The recoveries of the studied chlorinated benzenes decreased from 76–91% to 66–89% as the spiking level decreases from 100–1000 $\mu g\,g^{-1}$ to 1–10 $\mu g\,g^{-1}$.

In a recent study, Meissner *et al.* used SDE for the determination of fatty alcohol sulfates in cosmetics (toothpaste) by combining this technique with a hydrolysis treatment. However, the application of SDE to the analysis of nonylphenol polyethoxylates in sewage sludge by Lee *et al.* failed when compared with the more efficient supercritical fluid extraction technique.

In general, the published SDE methods for the analysis of non-fatty environmental samples involve longer extraction times (1–8 h) than those reported for aqueous samples (1–1.3 h). In addition, and contrary to that proposed by the theoretical model of Rijks *et al.*, the recoveries of less volatile compounds from non-fatty complex samples have been found to be independent of the vapour flow rates. However, it is important to note that in this study by Seidel ethanol was added to the water flask to improve the OCP recoveries, and that the possible effect of a co-distillation solvent was not included in the theoretical model.

Application of the SDE to the Analysis of Fatty Biological Samples and Food

Due to the high lipophility of some of the most toxic pollutants, such as OCPs, PCBs and PCDD/Fs, the classical procedures for the analysis of these pollutants in fatty samples were based on an exhaustive extraction of the lipids from the matrix. Subsequent removal of the co-extracted lipids has been widely recognized as the main problem with these kinds of methods, especially when analysing samples with high fat contents such as dairy products. Because of the characteristics of the SDE technique, the disruption of the strongly bound pollutant-matrix in these samples can be accomplished before SDE by degradation of the matrix components entrapping the target compounds. Treatment with 1–2 M sulfuric acid followed by ultrasonication in a bath and heating of the sample during the SDE process has been found to be one of the most efficient procedures for breaking down the matrix structure allowing steam distillation of the analytes. Furthermore, this acid treatment allowed a simultaneous clean up of the final extract as the matrix components form more polar products, which can then be easily separated from the non-polar analytes. According to the published results, most samples submitted to this kind of treatment did not require any additional clean up. Filek *et al.* report good recoveries for the SDE of OCPs from dairy products when using this type of acid pre-treatment: in the range 83–126% for powdered milk and human milk spiked at the 20.0–51.3 $ng\,g^{-1}$ level, and in the range of 73–111% for a certified dairy product (OCP levels ranging from 1.5 to 6.6 $ng\,g^{-1}$). However, the SDE method failed when it was used for the extraction of the endogenous PCBs from dairy products with different fat contents. According to the reference method, the PCBs detected by Ramos *et al.*, in 1998 ranged from 2 to 0.01 $ng\,g^{-1}$ in the investigated matrices. Nevertheless, most of the PCB congeners were found to be non-detectable with the SDE procedure and, when found at quantifiable levels, the reported concentrations were less than 26% of those determined by the reference method.

Rather similar results were reported by Seidel and Lindner in 1993 for the analysis of the OCPs in dairy products and human milk as none of the investigated compounds were found to be at a quantifiable level. However, no additional comparison with a reference method was included in this study, in which 10 g of sample was blended with water and ethanol. In this case, the alcohol, added as a co-distillation solvent, would also be able to disrupt the fat globule thereby allowing the SDE of the analytes. An important shortcoming of this kind of approach is the formation of large oil drops during the extraction, which increase the diffusion layer and hinder the SDE process. Filek *et al.* proposed blending of the sample with surfactants has been proposed as a possible solution for the case of fatty matrices without natural emulsifiers.

When no pre-treatment of the fatty sample was carried out, a co-distillation (total, according to Yoon *et al.*, or partial, according to Ramos *et al.*) of the lipids with the less volatile compounds occurred. Then, a post-treatment for isolation of the target compounds from the co-extracted matrix components was required. Following the implication of these results, it is rather surprising that neither pre- nor post-treatment of the sample was included

Table 3 SDE methods for the analysis of less volatile organic pollutants in fatty environmental samples (symbols as in Table 1)

Matrix	*Compound*	*Spiking level (ng g^{-1})*	*Pre-treatment*	*Solvent*	*Extraction time (h)*	*Post-treatment*	*Recovery (%)*	*RSD (%)*	*Ref.*
Fish tissue	PCBs	1700	Blended with 2.5 L water	Isooct/tol.(15)	7–14	NR	82–85	?	Veith *et al.* (1977)
Muscle, liver, kidney	PCBs, PCNs, ΣDDT[a]	5000	Blended with 0.1 L water	n-Heptane (10)	2	Concentration	67–100	1.0–20	Dunnivant *et al.* (1988)
Muscle, liver, kidney	PCBs, PCNs, ΣDDT[a]	1000	Blended with 0.1 L water	n-Heptane (10)	2	Concentration	65–85	?	
Dairy products,	Endogenous	–	10 g sample + water + ethanol	Petrobenzine (?)	1	NR	–	?	Seidel *et al.* (1993)
Human milk	OCPs								
Pumpkin seed	OCPs	100	10 g sample + water + ethanol + surfactant	Petrobenzine (?)	1	NR	65–89	6–12	
Certif. dairy product	OCPs	1.5–6.6	5 g sample + 80 mL 2 M H_2SO_4 + ultrasonic, 1 min + 1 mL ethanol + surfactant	Petrobenzine (20)	1.5	Concentration	73–111	?	Filek *et al.* (1995)
Powdered milk, Human milk	OCPs	20.0–51.3	5 g sample + 80 mL 2 M H_2SO_4 + ultrasonic, 1 min + 1 mL ethanol + surfactant	Petrobenzine (20)	1.5	Concentration	83–126	?	
Powdered milk	Endogenous PCBs	–	15 g sample + 60 mL 1 M H_2SO_4 + ultrasonic, 1 min	Dichlorometane (2)	60–90	SiO_2-HSO_4	< 26	?	Ramos *et al.* (1998)
Dairy products	PCBs	0.5	15 g sample + 60 mL 1 M H_2SO_4 + ultrasonic, 1 min	Dichlorometane (2)	60	SiO_2-HSO_4	< 10	?	
Herbal essential oils	OCPs	500–10 000	Blended with 0.05 L water	?	?	LLE (hexane : ethyl ether) + H_2SO_4	83–105	2.4–10	Rajendran *et al.* (1991)
	OPPs	500–10 000	Blended with 0.05 L water	?	?	LLE (hexane : ethyl ether) + H_2SO_4	72–116	0.5–10	

[a]DDT + DDE + TDE.

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in some of the first reported applications of SDE for the analysis of toxic aromatic compounds in biological matrices. The investigated samples included fish tissues, muscle, liver and kidney and, although satisfactory recoveries (67–100%) were reported for the spiked PCBs, PCNs and ΣDDT (i.e. DDT + DDE + TDE), it is important to note that the spiking level in these experiments ranged from 1000–5000 $ng\,g^{-1}$ (**Table 3**). Even at such a high level of concentration, the authors reported an evident dependence of SDE recoveries on the analyte concentration. In fact, Cooke *et al*. found that the PCB, PCN and ΣDDT recoveries from animal tissues decreased from 67–100% to 65–85% when the spiking level decreased from 5000 to 1000 $ng\,g^{-1}$. According to this trend, it can be concluded that the very low levels of the endogenous pollutants in environmental samples together with the typical complexity of the matrix would be the main reasons for the disappointing results reported for some SDE applications involving non-spiked fatty samples.

See also: **II/Extraction:** Analytical Extractions; Solid-Phase Extraction; Solid-Phase Microextractions; Supercritical Fluid Extraction. **Distillation:** Extractive Distillation.

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